Study of Electron-Phonon Interactions in

III-V Semiconductors

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Experimental Investigation of the Electron-Phonon Interaction in III-V Semiconductors

Introduction

A considerable amount of work has been done both experimentally[1] and theoretically[2] on the interaction of phonons with electrons in solids. Much of the work has centered on the attenuation of phonons in metals. In recent years many workers[3] have investigated both attenuation and gain in semiconductors, particularly CdS. In CdS the electron-phonon interaction is principally through the piezoelectric effect, whereas in non-piezoelectric semiconductors the interaction is principally through deformation potential coupling.

Calculations made by Spector[4] suggest that the electron-phonon interaction in non-piezoelectric semiconductors increases linearly with frequency in the high frequency region. This strong interaction would produce a high attenuation of a phonon wave unless the electrons in the solid are moving in the direction of the ultrasonic wave with a velocity exceeding that of the phonons. In this case the attenuation would be negative giving a gain in phonon energy. There are, however, other sources of attenuation besides the electron-phonon interaction. Among these are scattering from thermal phonons, impurity scattering, and

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crystal defect scattering. Thus even though a high attenuation might be measured in a material, the corresponding gain which should occur when the electron drift velocity exceeds the sonic velocity might not be as high as expected on the basis of the electron-phonon interaction alone.

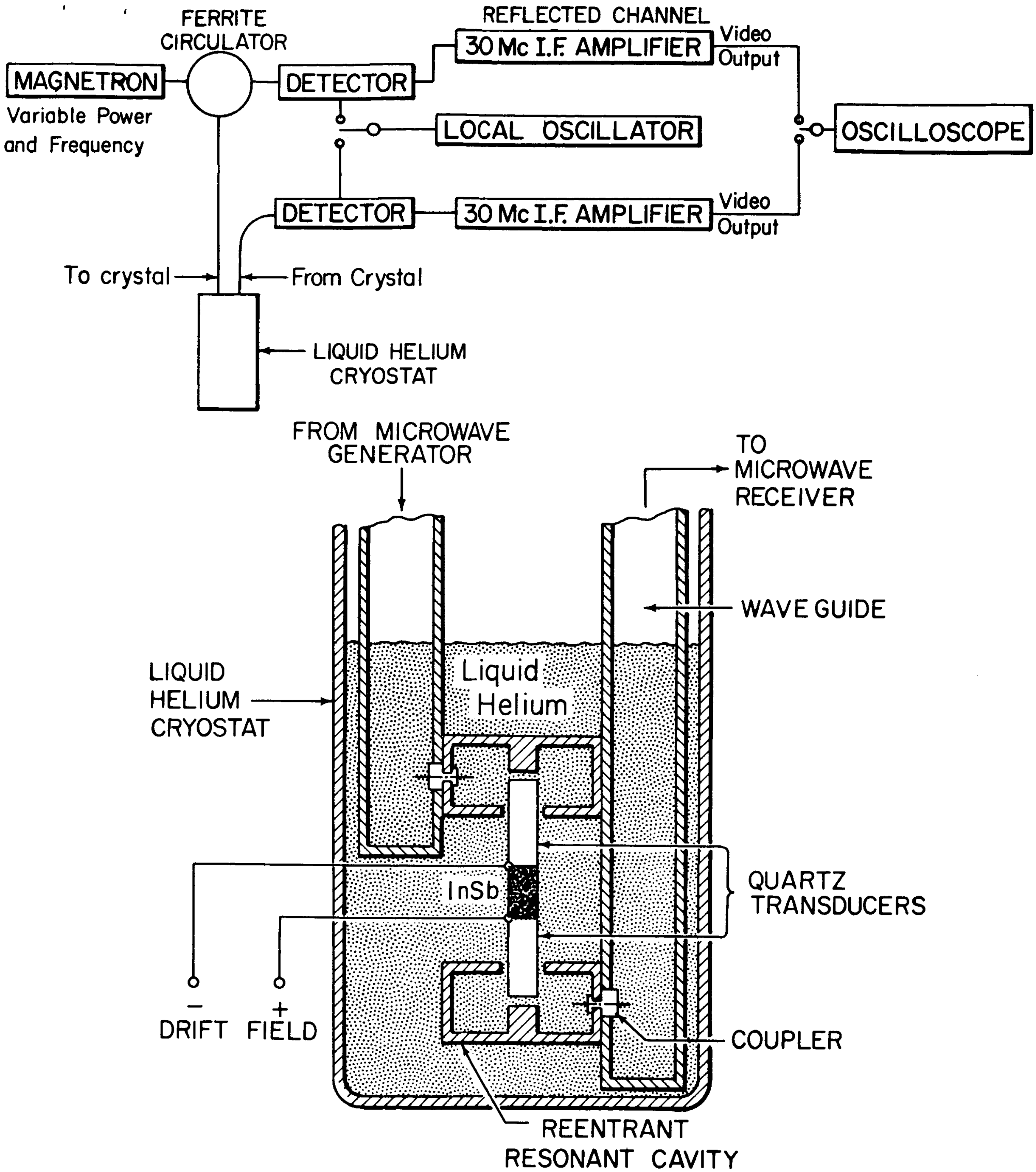
It is the purpose of this research program to investigate the various sources of attenuation in III-V semiconductor crystals and to investigate under what conditions amplification is possible. In this first quarterly progress report we discuss the experimental efforts at obtaining attenuation measurements in InSb at both 10 kmc and 10 mc frequencies.

1. 10 kmc Phonons

A. Crystal A

An Indium Antimonide single crystal of the highest purity obtainable from Cominco Products, Inc., was purchased and prepared for attenuation measurements. The crystal was an n type, boat-grown, zone refined crystal (which we shall refer to as crystal A) with a quoted carrier concentration, $N_e = 8.6 \times 10^{13}$ cm^{-3} and mobility $\mu_e = 5.7 \times 10^5$ cm^2 (volt sec)$^{-1}$ at 78°K. We determined the orientation of the (111) axis of the crystal to within $\pm$ 2° using a back reflection Laue x-ray diffraction camera. Using an ultrasonic cutter, the crystal was then cut into two 5/8" lengths with the cuts parallel to the (111) planes. These lengths of crystal were then cut into cylindrical rods 3 mm in diameter and 5/8" long again with the ultrasonic cutter maintaining the ± 2° accuracy of alignment between the (111) axis and the rod axis. These rods were then mounted with sealing wax into 3.2 mm diameter holes in the centers of 2 inch diameter, 5/8" thick glass disks. These disks were then optically polished so as to polish the ends of the rods. Optical interferometer methods were used on the disks during the polishing operations to insure that the end faces of the rods were held to within 6 seconds of parallelism which is equivalent to about 0.08 acoustic wavelengths in InSb.

The rods thus obtained from crystal A were tried for acoustic propagation of longitudinal waves at 10 kmc by several transducer methods. None of them were successful in obtaining any detectable acoustic propagation through the crystal. The basic equipment used to perform all of these tests is illustrated schematically in Fig. 1. All the experiments are done in liquid helium in order to minimize losses due to scattering from thermal phonons. The system uses a pulsed variable frequency magnetron whose energy is passed through a variable attenuator and then down a stainless steel waveguide into a tunable reentrant cavity. This cavity has a hole at the post end as shown in Fig. 1 where an x-cut quartz rod is inserted to serve as a piezoelectric transducer, converting the microwave electromagnetic energy into longitudinal acoustic waves in the quartz. The acoustic wave then travels through the quartz to the quartz-InSb bond where some of the acoustic energy is reflected and some is transmitted into the InSb. Another quartz rod is attached to the other end of the



SIMPLIFIED SCHEMATIC DIAGRAM OF MICROWAVE PHONON GENERATING AND DETECTING EQUIPMENT

Fig. 1

InSb and inserted into another identical reentrant resonant cavity to convert the acoustic wave by the inverse piezoelectric effect into a detectable microwave signal. A superheterodyne receiver system is used to detect this signal. It can also be switched over to one arm of a ferrite core circulator on the input waveguide to detect the signals which correspond to acoustic waves reflected back to the transmitting cavity. A standard signal generator is also fed into the receiver in order to calibrate the detected signal strengths.

The first method attempted involved the use of two quartz transducers on either end of the InSb rod. A method of bonding which had been found very successful for ultrasonic propagation in bonding tests between two quartz rods (resulting in nearly lossless bonds) consisted of pressing the two polished surfaces together without any bonding agent between the surfaces but with a ring of Epoxy cement* around the joint as illustrated in Fig. 2a. This failed in bonding quartz to InSb since the differences in coefficients of expansion caused the InSb to break each time.

The second method involved an attempt to overcome this difficulty. As is shown in Fig. 2b, a frame of Titanium was constructed such that it was glued to the sides of the quartz transducers but not to the InSb. The frame was glued on while the quartz transducers were held onto the InSb under pressure. The choice of Titanium lay in the fact that its coefficient of expansion lies between that of quartz (in the x-direction) and InSb. Thus by adjusting the length of the Titanium

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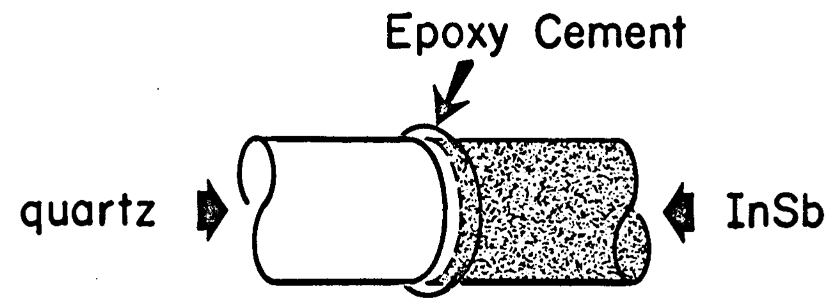
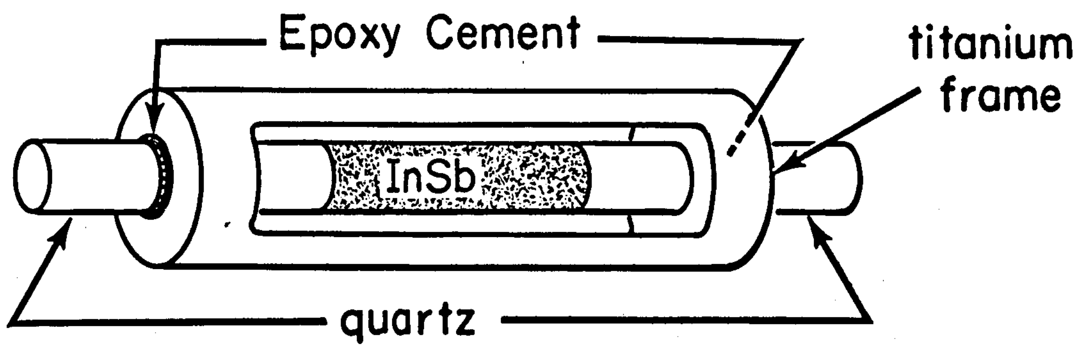
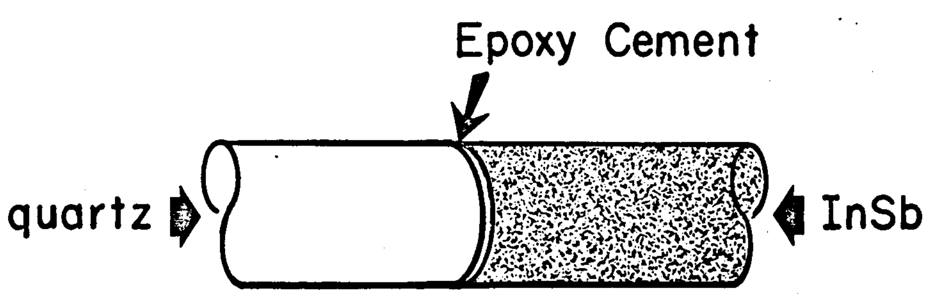


(a) Epoxy around joint but not between surfaces.



(b) Titanium frame holds rods together with no epoxy between the rod surfaces.



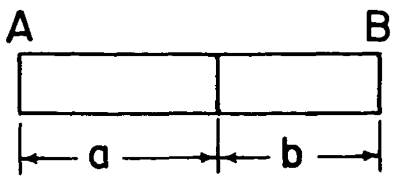
(c) Epoxy between the surfaces.

Fig. 2 TRANSDUCER BONDING METHODS

frame relative to the InSb and the quartz it is possible to match the total contractions of the frame and the rods. No transmission of ultrasonic waves through the InSb crystal was observed in this test although the equipment was working quite well. The receiver detected a whole series of reflected echoes from the transmitting cavity indicating that everything was working properly on up to the quartz-InSb interface. Similarly the receiving cavity was switched over to serve as transmitting cavity and similarly a series of echoes was observed although no transmitted pulses were detected. Thus the sound was highly attenuated either in the bonds or in the InSb itself. The sensitivity of the system is such that this represents a minimum attenuation of 40 db in the InSb and/or bonds.

The third method consisted of straightforward bonding of InSb to quartz using a very thin film of Epoxy Cement between the surfaces as illustrated in Fig. 2c. This method has also proved successful for near lossless transmission of 10 kmc phonons in bonding tests with two quartz rods (Fig. 3). The same procedure was followed as in the second method and again no acoustic transmission was observed through InSb.

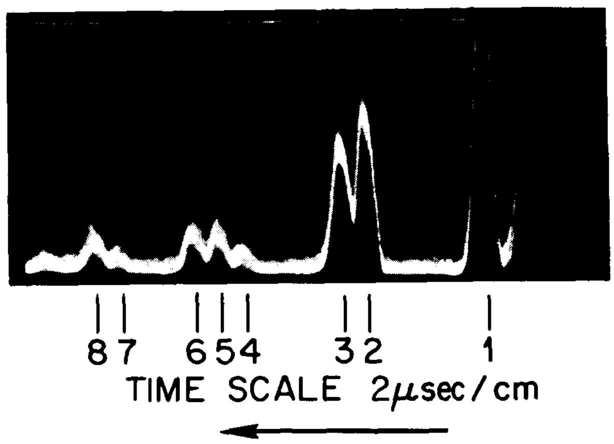
The fourth method amounted to simply inserting the InSb rod directly into the reentrant cavity, doing away with both quartz transducers. This attempt was based on the possibility that the piezoelectric coefficient of InSb would be large enough that it could act as its own transducer. This has the advantage of not involving any bonding problems. However, even though InSb lacks a center of



Transit time of the sound waves in the quartz crystals.

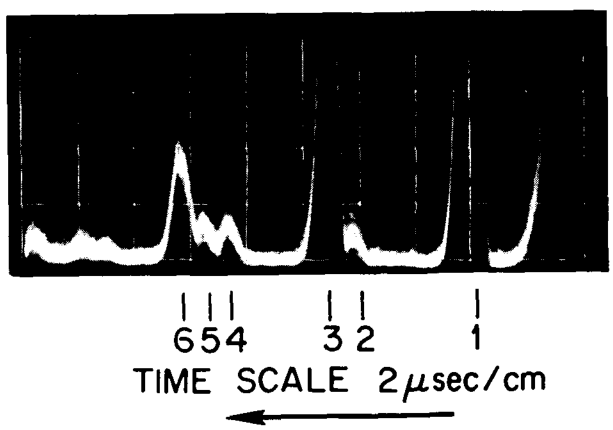
a = 2.6 microseconds

b = 2.2 microseconds



Transmit and receive at "B" end

1. $2b = 4.4\,\mu sec$
2. $4b = 8.8\,\mu sec$
3. $2b + 2a = 9.6\,\mu sec$
4. $6b = 13.3\,\mu sec$
5. $4b + 2a = 14.0\,\mu sec$
6. $2b + 4a = 14.8\,\mu sec$
7. $8b = 17.6\,\mu sec$
8. $6b + 2a = 18.4\,\mu sec$



Transmit into "B" end Receive at "A" end

1. $b + a = 4.8\,\mu sec$
2. $3b + a = 9.2\,\mu sec$
3. $b + 3a = 10.0\,\mu sec$
4. $5b + a = 13.6\,\mu sec$
5. $3b + 3a = 14.4\,\mu sec$
6. $b + 5a = 15.2\,\mu sec$

NOTE: The starting time of both traces does not show. The trailing edge that shows in both pictures is the trailing edge of the leakage pulse from the transmitter.

SOUND WAVE ECHOES IN BONDED QUARTZ CRYSTALS

Fig. 3

inversion symmetry and is therefore expected to be piezoelectric, the binding is largely covalent and one expects the piezoelectric coefficient to be small. No transmission or reflection of acoustic waves was observed in this test.

B. Crystal B

The lack of a detectable transmitted signal in the second and third methods led us to suspect that perhaps the bonds were not at fault but that the signal was highly attenuated in the InSb. In order to be satisfied that we were not suffering seriously from off axis propagation[5] due to poor alignment of the (111) axis and the polished surface normal ($\pm$ 2°), we decided to make a better alignment. We therefore obtained another boat grown InSb crystal referred to as crystal B which had a quoted carrier concentration, $N_e = 8.4 \times 10^{13}$ cm^{-3} and mobility $\mu_e = 5.0 \times 10^5$ cm^2 (volt sec)$^{-1}$ at 78°K.

The method used to obtain better alignment involved the use of a precision x-ray spectrogonimeter shown in Fig. 4. The x-rays were collimated onto the end of the InSb rod and the detector collimator was rotated to the correct position to pick up the first Bragg angle scattering of the copper K_α line from (111) planes in InSb. The goniometer holding the crystal was then rotated through its two degrees of freedom to maximize the x-ray counts in the geiger tube. This established the (111) axis as the bisecter of the source and detector collimator angle. Then the x-ray tube was removed from its location behind the

Fig. 4 - X-ray spectrogoniometer with crystal in place.

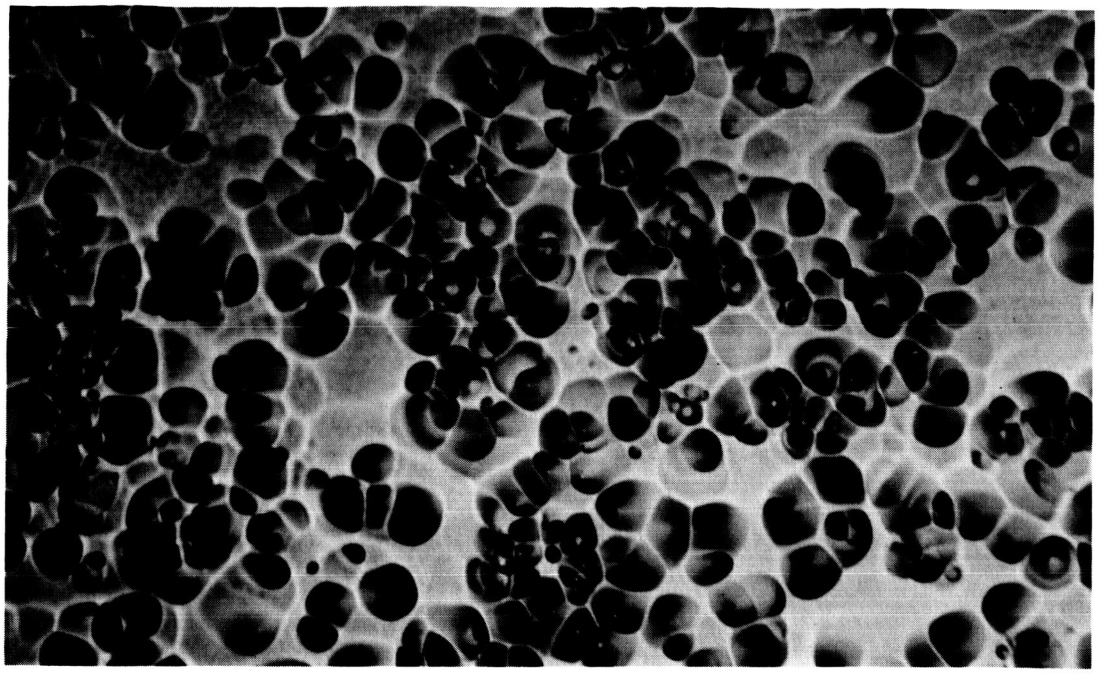
source collimator and replaced by a light source. A photomultiplier tube was placed behind the detector collimator instead of the geiger tube. The crystal goniometer was again rotated through its two degrees of freedom until the light collected from specular reflection from the polished surface was a maximum. The differences between the two sets of readings on the crystal goniometer represented the angular deviation, δ, between the (111) axis and the surface normal.

The InSb rod with its glass disc was then removed from the spectrogoniometer and repolished to remove this angular difference. The entire process was again repeated until δ was less then 3 minutes of arc. Finally the reverse end of the rod was polished by the method described earlier using optical interference to make the reverse end parallel to the other end to within 6 seconds of arc.

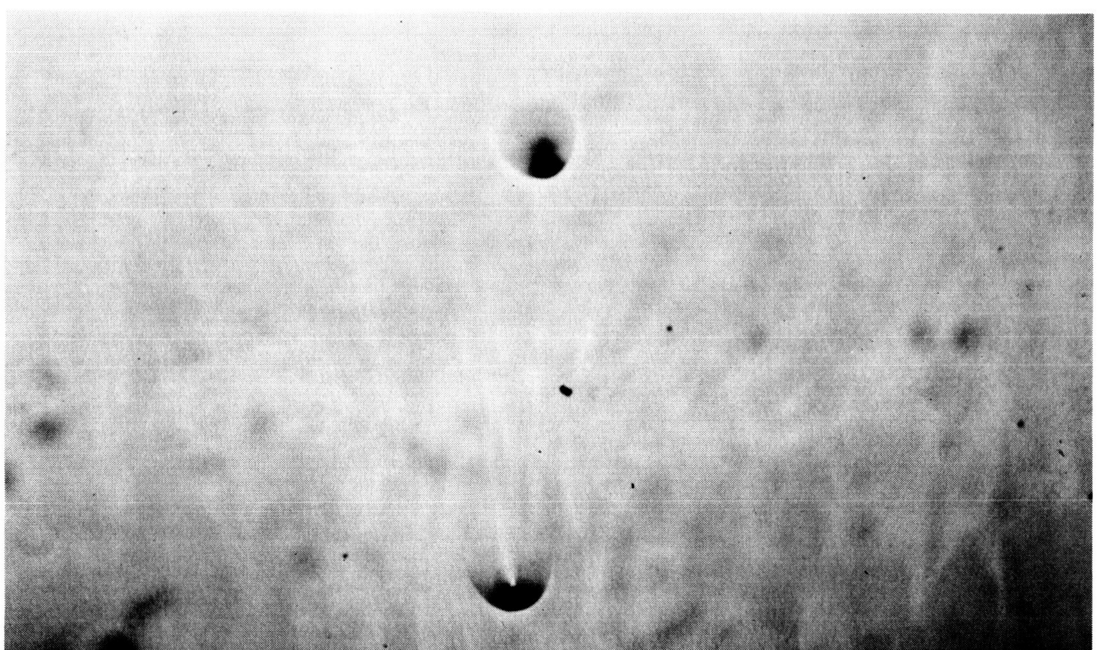
Two tests of crystal B using the third transducer method described for crystal A were performed with equally negative results. In the second test a voltage pulse was applied across the InSb during the microwave pulse in the hope of amplifying any signal if it were present. In order to expect any amplification the electron drift velocity, v_D, produced by the applied voltage pulse must exceed the sonic velocity v_S in InSb. Since $v_S = 3.88 \times 10^5$ cm/sec in InSb[6] we attempted to apply a voltage pulse such that $v_D = 2v_S = 7.76 \times 10^5$ cm/sec. To estimate the voltage required we must obtain the mobility at 4°K. According to data presented by Hilsum and Rose Innes[7], the Hall coefficient changes very little from 78°K to 4°K and the conductivity goes down by a factor of 33. Thus the mobility should go down by the same factor and we would expect therefore a mobility $\mu_e = 1.5 \times 10^4$ cm^2 (volt sec)$^{-1}$ at 4°K. Thus $E = v_o/\mu_e$ = 52 volts/cm requiring about 45 volts across a sample 0.86 cm long. The resistivity would be 4.9 Ωcm yielding a resistance of 60Ω for the 3 mm dia rod. These values do not agree with the values which we obtained when we applied pulses to the crystal. With pulses below 10 volts there was an apparent contact resistance of about 100,000Ω (which was not present in the range 77°K - 300°K). However above 10 volts this contact resistance broke down and the resistance dropped to 1 ohm such that up to 80 volts the resistance remained at this value. The leads had been soldered with pure Indium using ruby flux after cleaning the contact area with fine sandpaper.

The above mentioned experiments have left a number of unanswered questions which we are attempting to understand.

The success of the bonding techniques with quartz to quartz bonds leads us to suspect that the lack of observed acoustic propagation in InSb is characteristic of the InSb in some way. The possibility arises that there is considerable scattering from grown-in dislocations. Therefore we examined samples from crystal B for dislocations. This was done using a standard etchant solution, CP4A (3 parts 48% HF, 5 parts 68% HNO_3 and 3 parts glacial CH_3COOH) for 10 seconds. This was long enough to etch away completely any surface dislocations arising from abrasion, showing only the grown-in dislocation etch pits. A density of $10^6/cm^2$ was observed (Fig. 5a).



a - Crystal B, $10^6/cm^2$



b - Crystal C, $45/cm^2$

Fig. 5 - Dislocation etch pits produced in InSb crystals using CP4A etchant for 10 secs. Magnification 625X.

C. Crystal C

Although it possible to zone refine a boat-grown crystal to a higher purity than a pulled crystal the boat-grown crystal tends to have a much higher dislocation density. It was therefore decided to obtain a crystal which was grown by the Czochralski pulling method. This crystal will be referred to as crystal C. Crystal C had quoted values of $N_e = 3.5 \times 10^{14}$ and $\mu_e = 5 \times 10^5$ at 78°K. The same etchant was used for 10 seconds on crystal C as on crystal B and the photo of some of the resultant etch pits is shown in Fig. 5b. The density measured was $45/cm^2$. If the high attenuation was due to the high dislocation density, then this crystal should be much better. This crystal is being cut, aligned and polished in the same manner as crystal B and the same tests will be performed.

In order to better understand what was happening at 4°K when a voltage pulse was applied, measurements of the Hall coefficient and resistivity will be made in liquid helium. These measurements were made here at 300°K and at 77°K but the high contact resistances at 4°K made the measurements meaningless at that temperature. An ultrasonic soldering iron is being purchased in order to make better ohmic contacts for these measurements.

2. 10 mc Phonons

Equipment has now been set up for measurements of propagation of longitudinal waves in InSb. Preliminary measurements have been made at 300°K showing an attenuation of 0.54 db/cm in crystal C. An attempt was made to measure the attenuation at 77°K but the transducer bond broke before any measurement could be made. However, it was evident before the bond broke, that the attenuation was much less than at 300°K. No re-run had been made before the writing of this report.

Preparations are under way to apply a voltage across the InSb during the 10 mc pulse to look for changes in attenuation with voltage. A complete description of the equipment used for these measurements will be given in the next quarterly progress report.

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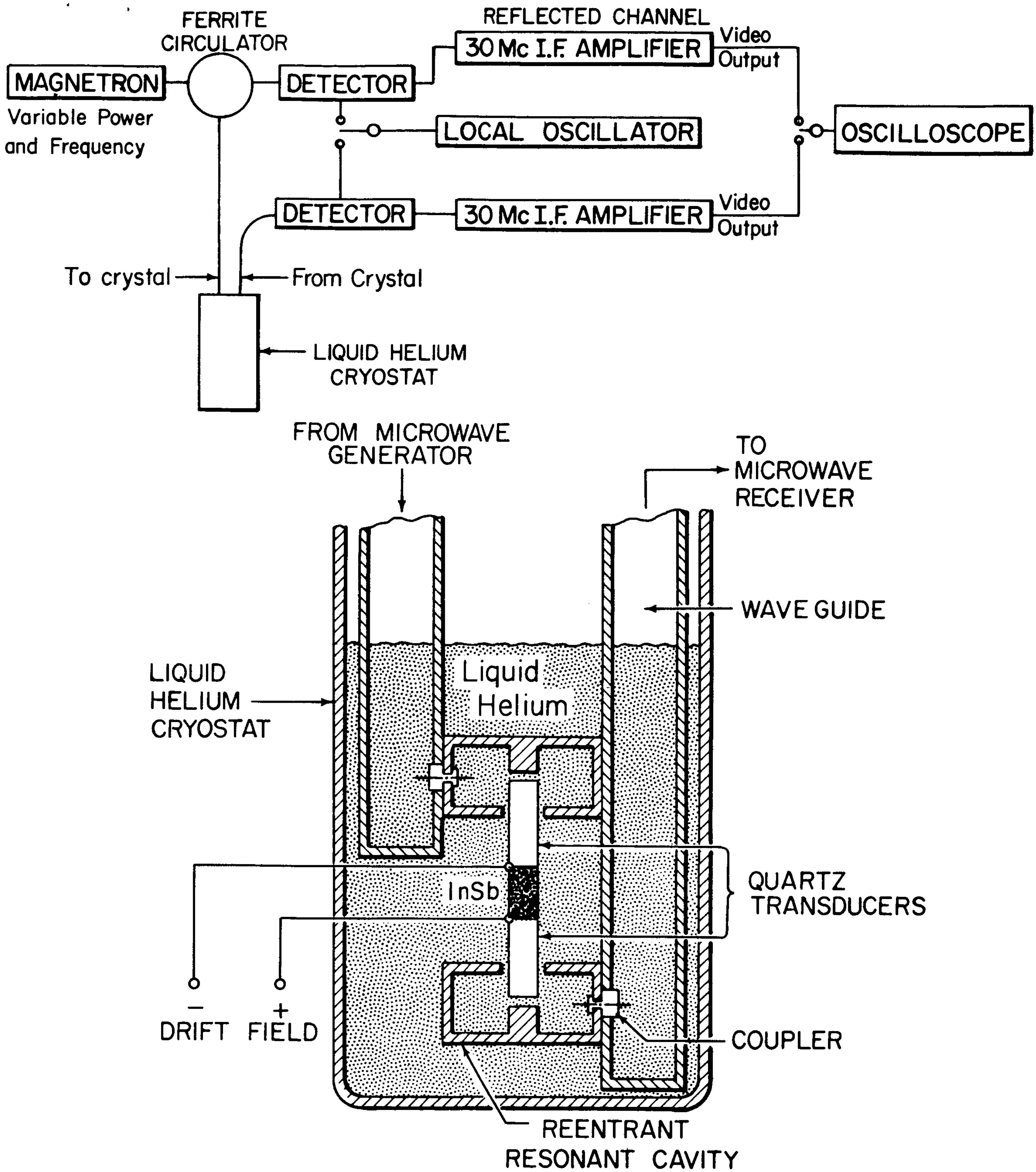

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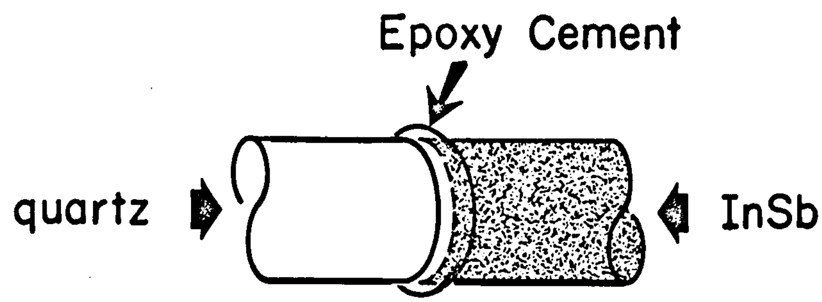
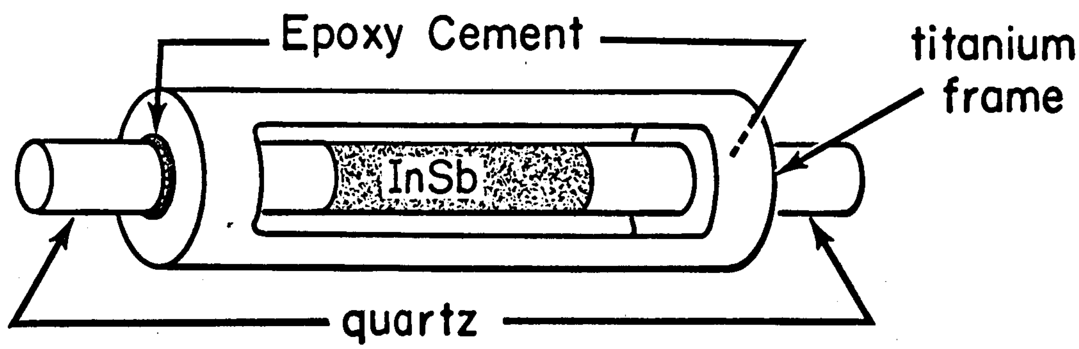


(a) Epoxy around joint but not between surfaces.



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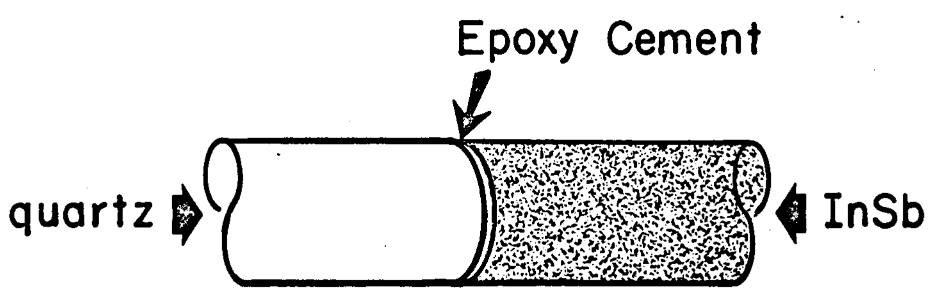

(c) Epoxy between the surfaces.

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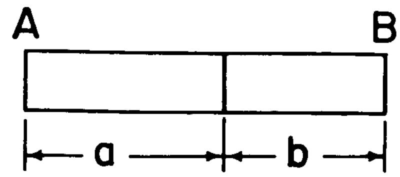

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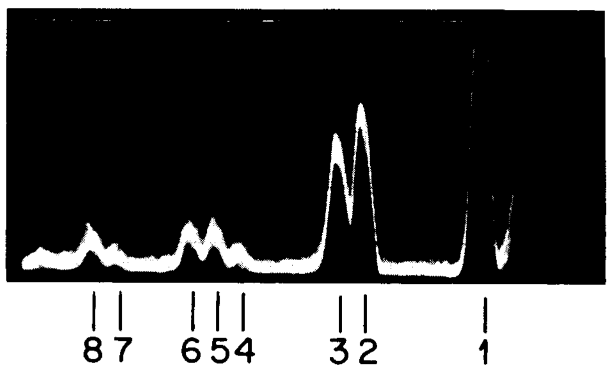

TIME SCALE 2 μsec/cm

Transmit and receive at "B" end

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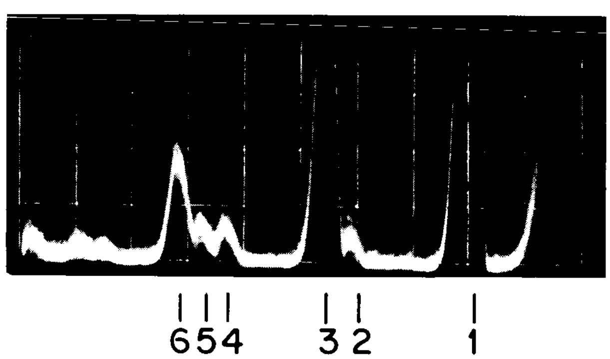
7. 8b = 17.6 μsec
8. 6b + 2a = 18.4 μsec



TIME SCALE 2 μsec/cm

Transmit into "B" end
Receive at "A" end

1. b + a = 4.8 μsec
2. 3b + a = 9.2 μsec
3. b + 3a = 10.0 μsec
4. 5b + a = 13.6 μsec
5. 3b + 3a = 14.4 μsec
6. b + 5a = 15.2 μsec

NOTE: The starting time of both traces does not show. The trailing edge that shows in both pictures is the trailing edge of the leakage pulse from the transmitter.

SOUND WAVE ECHOES IN BONDED QUARTZ CRYSTALS

Fig. 3

inversion symmetry and is therefore expected to be piezoelectric, the binding is largely covalent and one expects the piezoelectric coefficient to be small. No transmission or reflection of acoustic waves was observed in this test.

B. <u>Crystal B</u>

The lack of a detectable transmitted signal in the second and third methods led us to suspect that perhaps the bonds were not at fault but that the signal was highly attenuated in the InSb. In order to be satisfied that we were not suffering seriously from off axis propagation[5] due to poor alignment of the (111) axis and the polished surface normal ($\pm$ 2°), we decided to make a better alignment. We therefore obtained another boat grown InSb crystal referred to as crystal B which had a quoted carrier concentration, $N_e = 8.4 \times 10^{13}$ cm^{-3} and mobility $\mu_e = 5.0 \times 10^5$ cm^2 (volt sec)$^{-1}$ at 78°K.

The method used to obtain better alignment involved the use of a precision x-ray spectrogonimeter shown in Fig. 4. The x-rays were collimated onto the end of the InSb rod and the detector collimator was rotated to the correct position to pick up the first Bragg angle scattering of the copper K_α line from (111) planes in InSb. The goniometer holding the crystal was then rotated through its two degrees of freedom to maximize the x-ray counts in the geiger tube. This established the (111) axis as the bisecter of the source and detector collimator angle. Then the x-ray tube was removed from its location behind the

Fig. 4 - X-ray spectrogoniometer with crystal in place.

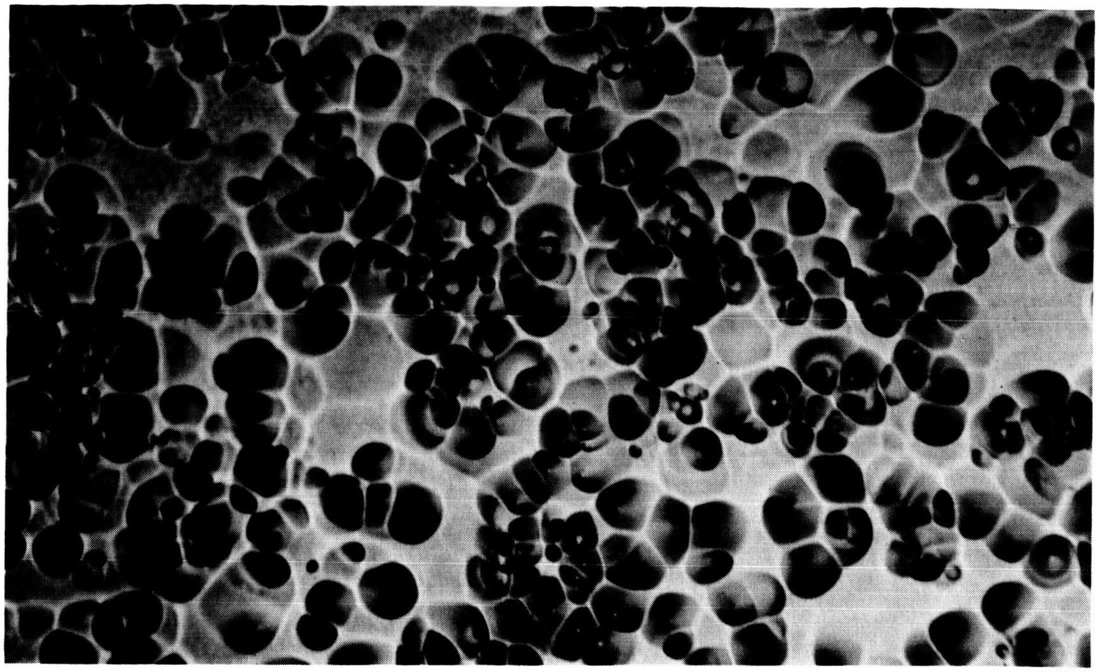
source collimator and replaced by a light source. A photomultiplier tube was placed behind the detector collimator instead of the geiger tube. The crystal goniometer was again rotated through its two degrees of freedom until the light collected from specular reflection from the polished surface was a maximum. The differences between the two sets of readings on the crystal goniometer represented the angular deviation, δ, between the (111) axis and the surface normal.

The InSb rod with its glass disc was then removed from the spectrogoniometer and repolished to remove this angular difference. The entire process was again repeated until δ was less then 3 minutes of arc. Finally the reverse end of the rod was polished by the method described earlier using optical interference to make the reverse end parallel to the other end to within 6 seconds of arc.

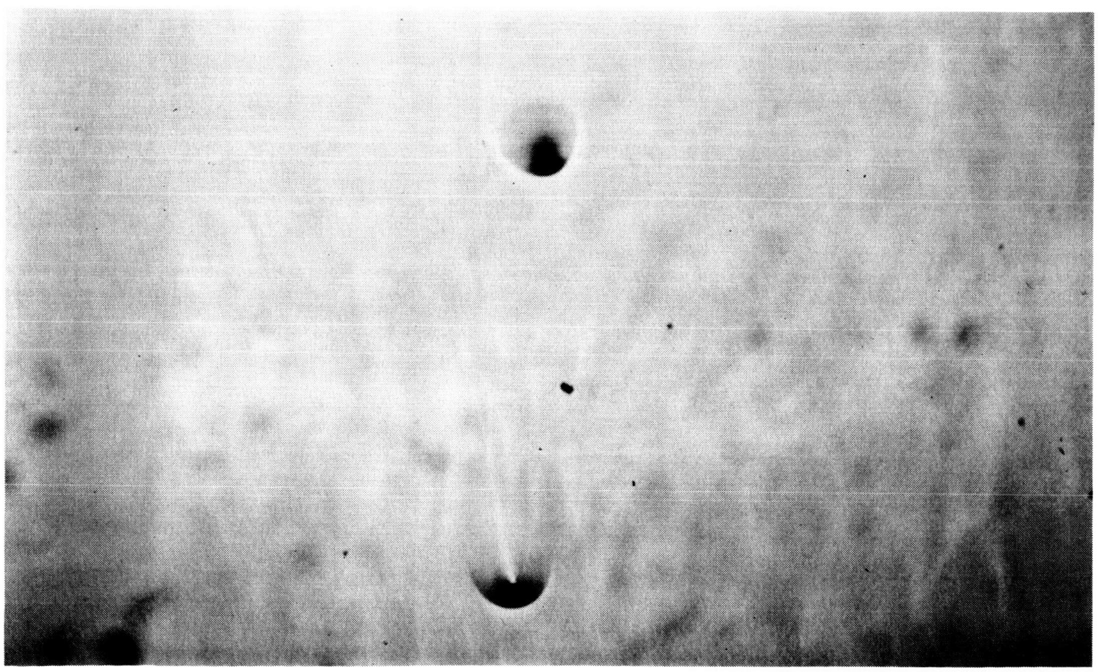
Two tests of crystal B using the third transducer method described for crystal A were performed with equally negative results. In the second test a voltage pulse was applied across the InSb during the microwave pulse in the hope of amplifying any signal if it were present. In order to expect any amplification the electron drift velocity, v_D, produced by the applied voltage pulse must exceed the sonic velocity v_S in InSb. Since $v_S = 3.88 \times 10^5$ cm/sec in InSb[6] we attempted to apply a voltage pulse such that $v_D = 2v_S = 7.76 \times 10^5$ cm/sec. To estimate the voltage required we must obtain the mobility at 4°K. According to data presented by Hilsum and Rose Innes[7], the Hall coefficient changes very little from 78°K to 4°K and the conductivity goes down by a factor of 33. Thus the mobility should go down by the same factor and we would expect therefore a mobility $\mu_e = 1.5 \times 10^4$ cm^2 (volt sec)$^{-1}$ at 4°K. Thus $E = v_o/\mu_e = 52$ volts/cm requiring about 45 volts across a sample 0.86 cm long. The resistivity would be 4.9 Ωcm yielding a resistance of 60Ω for the 3 mm dia rod. These values do not agree with the values which we obtained when we applied pulses to the crystal. With pulses below 10 volts there was an apparent contact resistance of about 100,000Ω (which was not present in the range 77°K - 300°K). However above 10 volts this contact resistance broke down and the resistance dropped to 1 ohm such that up to 80 volts the resistance remained at this value. The leads had been soldered with pure Indium using ruby flux after cleaning the contact area with fine sandpaper.

The above mentioned experiments have left a number of unanswered questions which we are attempting to understand.

The success of the bonding techniques with quartz to quartz bonds leads us to suspect that the lack of observed acoustic propagation in InSb is characteristic of the InSb in some way. The possibility arises that there is considerable scattering from grown-in dislocations. Therefore we examined samples from crystal B for dislocations. This was done using a standard etchant solution, CP4A (3 parts 48% HF, 5 parts 68% HNO_3 and 3 parts glacial CH_3COOH) for 10 seconds. This was long enough to etch away completely any surface dislocations arising from abrasion, showing only the grown-in dislocation etch pits. A density of $10^6/cm^2$ was observed (Fig. 5a).



a - Crystal B, $10^6/cm^2$



b - Crystal C, $45/cm^2$

Fig. 5 - Dislocation etch pits produced in InSb crystals using CP4A etchant for 10 secs. Magnification 625X.

C. Crystal C

Although it possible to zone refine a boat-grown crystal to a higher purity than a pulled crystal the boat-grown crystal tends to have a much higher dislocation density. It was therefore decided to obtain a crystal which was grown by the Czochralski pulling method. This crystal will be referred to as crystal C. Crystal C had quoted values of $N_e = 3.5 \times 10^{14}$ and $\mu_e = 5 \times 10^5$ at 78°K. The same etchant was used for 10 seconds on crystal C as on crystal B and the photo of some of the resultant etch pits is shown in Fig. 5b. The density measured was $45/cm^2$. If the high attenuation was due to the high dislocation density, then this crystal should be much better. This crystal is being cut, aligned and polished in the same manner as crystal B and the same tests will be performed.

In order to better understand what was happening at 4°K when a voltage pulse was applied, measurements of the Hall coefficient and resistivity will be made in liquid helium. These measurements were made here at 300°K and at 77°K but the high contact resistances at 4°K made the measurements meaningless at that temperature. An ultrasonic soldering iron is being purchased in order to make better ohmic contacts for these measurements.

2. 10 mc Phonons

Equipment has now been set up for measurements of propagation of longitudinal waves in InSb. Preliminary measurements have been made at 300°K showing an attenuation of 0.54 db/cm in crystal C. An attempt was made to measure the attenuation at 77°K but the transducer bond broke before any measurement could be made. However, it was evident before the bond broke, that the attenuation was much less than at 300°K. No re-run had been made before the writing of this report.

Preparations are under way to apply a voltage across the InSb during the 10 mc pulse to look for changes in attenuation with voltage. A complete description of the equipment used for these measurements will be given in the next quarterly progress report.

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